Storage of Activated Nickel-Hydrogen Cells and Batteries

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14. ABSTRACT

The preferred methods for storing activated nickel-hydrogen cells and batteries are described, along with the mechanisms for chemical and electrochemical processes that can occur during storage to influence the performance and dictate the optimum storage conditions.

15. SUBJECT TERMS

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1. Introduction

Nickel-hydrogen battery cells are subject to a number of chemical and electrochemical processes that can influence their behavior as a result of long-term storage after activation with electrolyte. The behavior of these cells during storage is strongly influenced by the precharge in the cell, as well as the voltage at which the cell is maintained during storage. The precharge refers to the electrode that has some remaining capacity after the other electrode has been fully discharged, and comes in two distinct flavors.

1. Nickel precharge means that the fully discharged cell contains no remaining hydrogen gas, and the nickel electrodes still contain some electrochemically active charge that could be discharged if there were more hydrogen available. The ampere-hours of electrochemically active charge in the nickel electrodes gives the quantity of nickel precharge, which is often expressed as a percentage of the nameplate cell capacity.

2. Hydrogen precharge means that the fully discharged cell contains no remaining active capacity in the nickel electrodes. In this condition, excess hydrogen gas remains within the fully discharged cell. The ampere-hours corresponding to this excess hydrogen gas gives the quantity of hydrogen precharge, which may be expressed simply as a pressure, or as a percentage of the nameplate cell capacity.

Most nickel-hydrogen cells manufactured today are made with a nickel precharge of approximately 7–20% of the nameplate cell capacity, depending on the cell design and the cell manufacturer. This precharge condition has been selected because the cells have been found to be much more stable during passive storage in the fully discharged state than are hydrogen precharged cells. This stability is because the precharge in the nickel electrode holds the potential of both the nickel electrode and hydrogen electrodes at 1.1–1.3 V relative to the hydrogen potential. At these potentials, few processes occur to alter the performance of the nickel or platinum catalyst electrodes. The few processes that do occur during storage to alter cell performance occur very slowly.

This report provides a summary of the chemical and electrochemical processes that are known to occur within the nickel-hydrogen cell during various methods of storage that have been used, with either nickel or hydrogen precharge. Recommendations are also provided indicating the preferred methods of cell or battery storage and maintenance for both nickel and hydrogen precharged conditions.

2. Chemical Processes in Stored Nickel-Hydrogen Cells

The chemical processes that occur in nickel-hydrogen cells are strongly dependent on the existing type of precharge within the cell. This strong dependence occurs because nickel precharge provides a highly oxidizing potential at the cell electrodes, while hydrogen provides a highly reducing potential at both the cell electrodes. There is approximately a 1.1- to 1.3-V potential difference between these two potential environments, thus the chemistry is dramatically different.
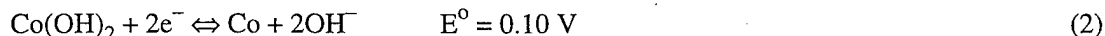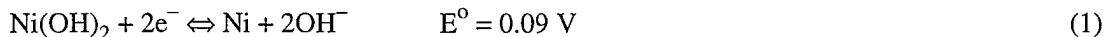
2.1 Hydrogen Precharge

When a nickel-hydrogen cell contains any measurable quantity of hydrogen gas, the hydrogen will hold the potential of the platinum catalyst electrode at the equilibrium hydrogen potential (which exhibits a Nernstian dependence on hydrogen pressure). The hydrogen gas will also react with the high-surface-area nickel sinter in the nickel electrodes, also tending to drive these somewhat catalytic surfaces towards the hydrogen potential. In this situation, the metal sinter in the nickel electrodes acts as a hydrogen electrode, and thus will go to essentially the same potential as the platinum electrode if the discharged cell is open-circuited. Under fully discharged conditions the open-circuit cell voltage will be observed to settle at a near-zero volt value. If the cell contains appreciable charge, this process will continue as a parasitic self-discharge of the cell capacity until either the hydrogen is depleted or the nickel electrode capacity becomes depleted.

The shorting junction between the active material and the hydrogen electrode formed by the nickel sinter can gradually discharge the residual charge in the nickel electrodes, assuming a sufficient amount of hydrogen gas. This electrocatalytic self-discharge process will eventually form an active material layer of significant thickness that is at or near the hydrogen potential. The long-term stability of the nickel electrode active material at hydrogen potentials is thus critical to the issue of open-circuit storage when a hydrogen precharge is present.

2.1.1 Low Potentials

When the cell is either held at potentials near zero volts (*vs.* hydrogen) or allowed to settle to low voltages in an open-circuited condition, the following reactions can occur in the nickel electrode active material that exists in the low-voltage layer in contact with the sinter. The thickness of this layer will typically increase with time spent at low potentials because the residual charge in the nickel electrode gradually depletes.

$$Ni(OH)_2 + 2e^- \Leftrightarrow Ni + 2OH^- \qquad E^o = 0.09 \text{ V} \qquad (1)$$

$$Co(OH)_2 + 2e^- \Leftrightarrow Co + 2OH^- \qquad E^o = 0.10 \text{ V} \qquad (2)$$

The cobalt in the active material exists as a co-deposited additive in the active material structure to improve its conductivity and performance. Reactions 1 and 2 will occur relatively slowly, but can cause modified layers around the sinter surfaces that are relatively thick (microns) in time periods of weeks to months. The thicker this modified layer becomes, the more likely it is to affect cell performance, and the more difficult it will be to disperse the layer and recover performance.

The metallic products of reactions 1 and 2 are very finely divided, and thus highly active to oxidation if at some later time the voltage is increased above 0.10 V. This situation arises whenever a stored cell is recharged as it comes out of storage. The reverse of reactions 1 and 2 will result in finely divided nickel and cobalt hydroxides, as opposed to the co-deposited active material that was initially present. The nickel hydroxide is relatively stable, and will simply ripen into larger crystallites that will undergo oxidation to NiOOH during recharge. The cobalt hydroxide, however, is relatively soluble in alkaline electrolyte, and can dissolve in the electrolyte to produce dicobaltite ions by reaction 3.

$$Co(OH)_2 + OH^- \Leftrightarrow HCoO_2^- + H_2O \tag{3}$$
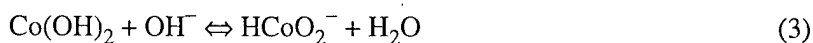
The cobalt hydroxide can also undergo oxidation by reaction 4 at a potential 1 V above the hydrogen potential.

$$Co(OH)_2 + OH^- \Leftrightarrow Co(OH)_3 + e^- \qquad E^\circ = 1.00 \text{ V} \tag{4}$$
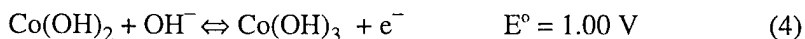
Since some of the cobalt in the modified layer at the sinter surfaces is converted into a soluble product that will diffuse away from the layer, the net effect is loss of cobalt additive from the layer of active material in contact with the nickel sinter in the nickel electrodes. The cobalt lost from this layer is deposited in various oxidized forms elsewhere in the nickel electrode, or, in some cases, on the surfaces of other cell components.

After the layered cobalt depletion described above, the nickel electrodes are more difficult to recharge efficiently, store less capacity, have poorer conductivity, and are not capable of discharging all their stored capacity. The net result is reduced cell capacity, in some cases by up to 20–30%. The only way to recover capacity is to somehow break-up the layered structure at the sinter surface. This can be done by repeated cycling, which mixes the layered material through repeated expansion and contraction, and through oxygen evolution. This recovery process is most effective when the active material is cycled into the high-volume gamma-NiOOH phase, which typically involves significant overcharge and low-temperature operation. Recovery can be simple and essentially complete when quite thin modified layers are involved. However, if cobalt-depleted layers form that are a significant fraction of the thickness of the active material deposits (10–20 μm), it can be virtually impossible to fully recover and maintain capacity.

Because of these degradation processes, it is very important to avoid storage of hydrogen-precharged cells at low potentials.

2.1.2 High Potentials

Storage of hydrogen-precharged cells at high potentials (i.e., partially charged) is the preferred storage method. At voltages above 1.0 V, the cobalt is stable in the trivalent state. At open-circuit voltages above about 1.2 V, the various phases of nickel oxyhydroxide are stable. Thus, little degradation to the active material deposits in the nickel electrode should occur if the cell is maintained in a partially charged state. However, the cell is highly active in this condition, and will undergo continuous self-discharge, heat generation, and evolution of oxygen. Cells in this storage state must be monitored and batteries periodically top-charged or trickle charged. The continuous throughput of charge causes slow degradation from overcharge and from corrosion of the nickel sinter at the highly oxidizing potential of the charged nickel electrode. This type of storage is similar, in terms of its effect on life, to charged maintenance during periods of non-cycling if a cold environment can be maintained.

2.2 Nickel Precharge

Cells having a nickel precharge contain an oxidizing atmosphere consisting of oxygen gas given off by the partially charged nickel electrode. The partially charged nickel electrode active material is unstable relative to the evolution of oxygen, and thus will slowly give off oxygen gas until a steady-state pressure is achieved. With 10–15% nickel precharge, the oxygen gas pressure found in a cell is typically less than 1 atmosphere. Any residual hydrogen in the cell is rapidly consumed by recombination with oxygen. During open-circuit storage, the platinum catalyst electrode is held at the equilibrium oxygen potential, which is typically 0.1–0.2 V below the potential of the nickel electrode, producing an open-circuit cell potential of 0.1 to 0.2 V. Thus, the nickel precharge effectively holds the potential of both electrodes at or near the highly oxidizing potential of the partially charged nickel electrode during open-circuit storage.

A number of chemical processes can take place in the nickel and platinum electrodes during long-term storage in this oxidizing condition. The nickel sinter in the nickel electrode as well as the platinum catalyst in the platinum electrode are unstable to oxidation. The corrosion of the nickel sinter, while taking place, is quite slow due to the passivation layers that form on the nickel metal surface. The platinum oxides that form on the platinum catalysts, however, are slightly soluble in alkaline electrolyte. Dissolution of platinum oxide occurs to establish a low equilibrium concentration of platinate ions in the electrolyte by reaction 5.

$$PtO_2 + 2H_2O + 2OH^- \Leftrightarrow Pt(OH)_6^- \qquad (5)$$
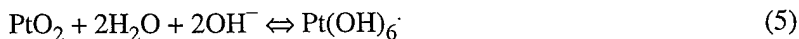
Platinate ions are capable of complexing with adjacent cobalt and nickel sites in the charged active material to form a compound containing no more than one platinate species per charged nickel and cobalt site in the lattice (Reaction 6). This compound forms within the precharged active material regions, which, in a nickel precharged cell, consist entirely of the lower potential gamma-nickel oxyhydroxide phase. This platinate complex is stable as long as it is not discharged and as long as the electrolyte contains dissolved platinate ions. The platinate complex discharges at a potential about 0.12 V lower than the potential at which uncomplexed active material discharges, as indicated in Figure 1. If it is discharged, recharge within several hours can regenerate a stable complex. If

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recharge is not done within several hours, the platinate ion will diffuse from the lattice and recharge will occur in a normal manner.

$$Pt(OH)_6^= + CoOOH + NiOOH \Leftrightarrow CoO\cdot Pt(OH)_6\cdot NiO + 2OH^- \qquad (6)$$
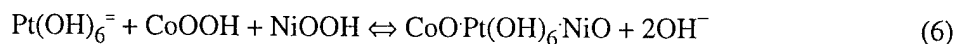
The platinate complex formed by reaction 6 does, in fact, have a significant effect on cell performance. It significantly reduces the recharge potential, improves the recharge efficiency, and results in higher cell capacity once the cell is brought out of storage and recharged. Unfortunately, the complex is not stable, and the beneficial effects on performance are typically seen for only the first several charge/discharge cycles. As soon as the cell is recharged the platinum electrode goes to the hydrogen potential, which causes the platinate ions in solution to be plated back onto the platinum electrode. The platinate-depleted solution will gradually cause the platinate complex in the nickel electrode to dissociate and feed the platinum back onto the platinum electrode by a plating process. The platinum plated back onto the platinum electrode will not be in the active, high-surface-area form of the original catalyst, but will have a much lower surface area. Thus, the only long-term effect of this entire process that has been seen to date is a small reduction in the activity of the platinum electrode. Fortunately, the platinum catalysts used in nickel-hydrogen cells have over an order-of-magnitude more catalyst than is required for adequate operation.
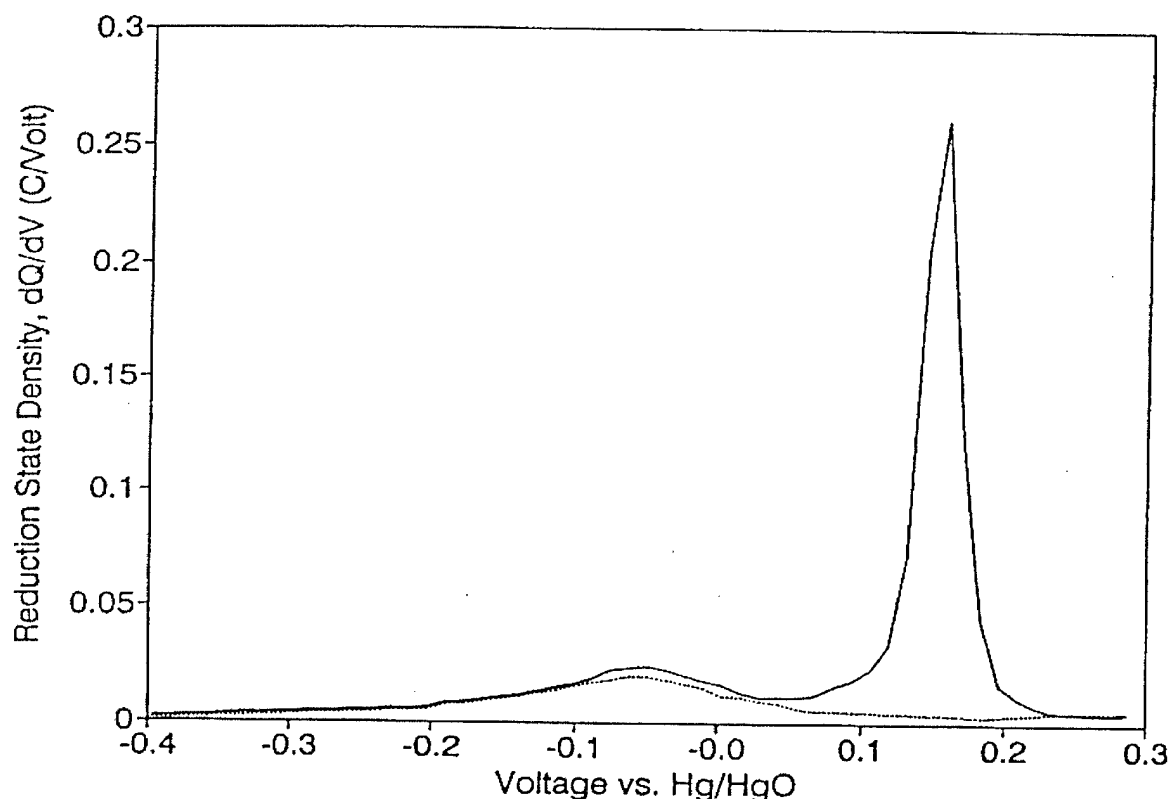

Figure 1.   Electrochemically reducible state density vs. potential for nickel electrodes, Solid line from nickel precharged cell and dashed line for a discharged new electrode. Total charge in the peak from the platinum complex near 0.15 V is about 5 mAh/cm^2.

3. Precharge Determination in Nickel-Hydrogen Cells

It is clear from the discussions above that it is critical to know the type of precharge in nickel-hydrogen cells at the time they are put into storage. Several methods are indicative of precharge type, and will be discussed here along with the preferred method for definitively indicating precharge type.

1. **Residual Capacity and Second Plateau.** The nickel electrode contains approximately 10% of its capacity in the form of residual capacity that is only discharged at rates less than C/2, often on a lower voltage plateau near 1.0 V. Thus, a hydrogen-precharged cell would exhibit all this residual capacity, while a nickel-precharged cell would exhibit none or only part of the typical residual capacity. However, because the precise amount of residual capacity can vary significantly over differing cycling conditions, this method only provides a rough indication of precharge type, rather than being definitive.

2. **Voltage Recovery After Letdown.** If a nickel precharged cell is resistively let down to less than 0.01 V, then allowed to recover to its open-circuit potential, it will develop a potential of about 0.2 V. This is the potential between the partially charged nickel electrode and the oxygen potential on the platinum catalyst electrode. A hydrogen-precharged cell will either stay below 0.01 V, or if it has some residual nickel capacity, will rise in voltage for a time, then go back towards zero volts as self-discharge occurs. The main problem with this method for determining precharge type is that the voltage with hydrogen precharge passes through the stable region for nickel precharge. Thus, a good determination requires assurance that the voltage has achieved a steady level. This can take significant time in many instances.

3. **Reversal Response.** If a fully discharged nickel-hydrogen cell with nickel precharge is reversed for 5 min at a C/40 rate, a negative-going voltage response falling below –0.2 V will be seen at the 5-min point, as indicated in Figure 2. If the cell has hydrogen precharge, the voltage after 5 min will be above –0.1 V, and will typically be rising towards zero volts, as indicated in Figure 3. These two dramatically different signatures are easily recognized responses corresponding to the differing precharge environments. This test has been found to work best when it is done after a full recharge and C/2 discharge cycle having no open-circuit time between charge and discharge, then an immediate resistive letdown to 0.005 V using a 0.5-ohm resistor (50 Ah cell).
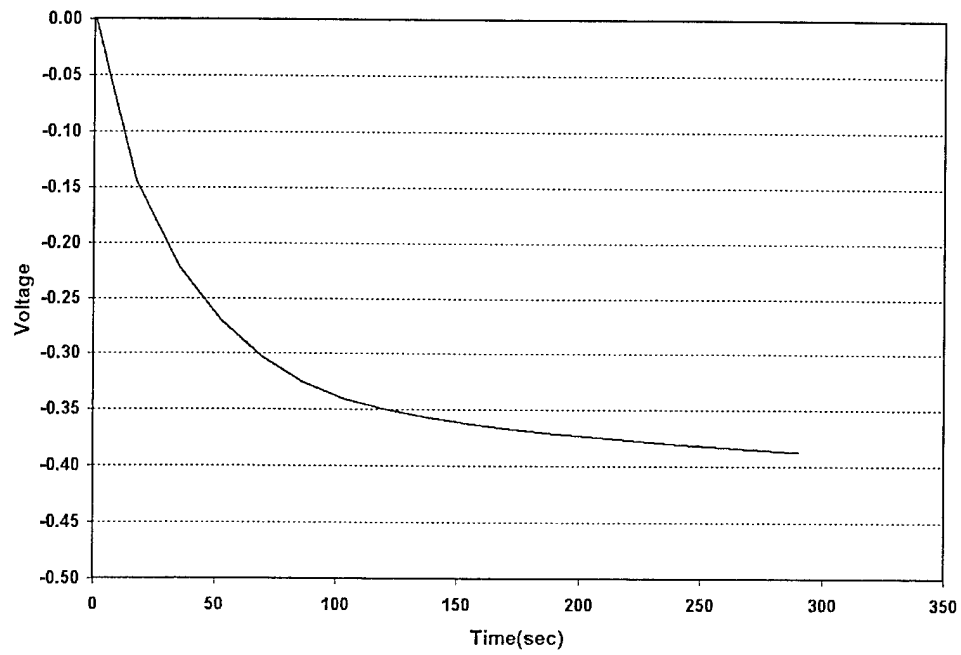
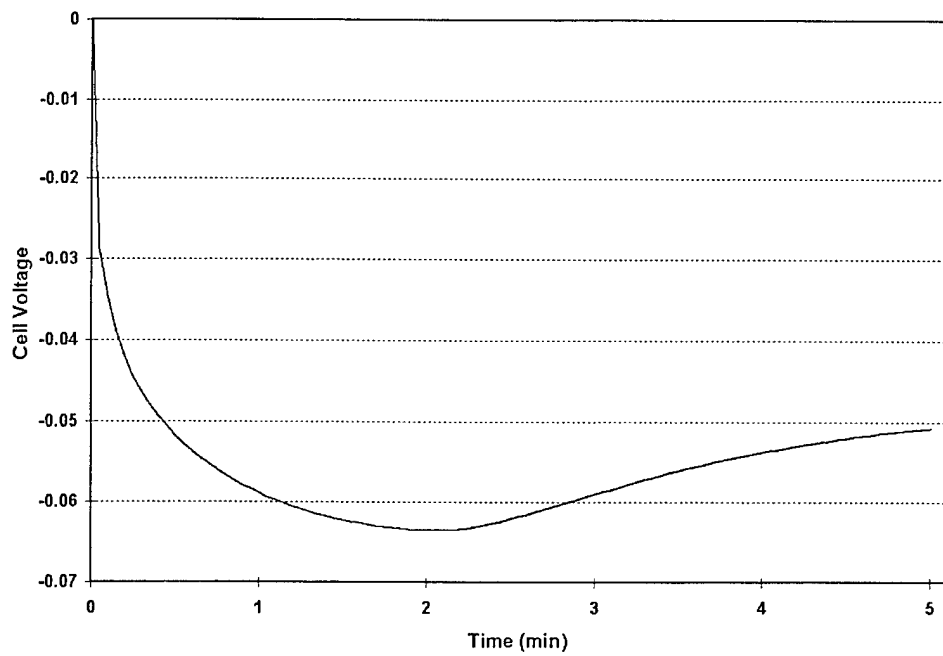Figure 2. Response of nickel-precharge cell to a 5-min reversal.

Figure 3. Response of hydrogen-precharge cell to a 5-min reversal.

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4. Recommended Battery Storage Procedures and Methods

It is recommended that cells be fabricated with a nickel precharge. This allows the cells and batteries built from the cells to be stored in a fully discharged state, as long as there remains verified active nickel precharge within all the cells. The following procedure is suggested to prepare cells and batteries for discharged storage in a manner that maintains optimal activity for the nickel precharge, and verifies that each cell has some active precharge before storage begins.

4.1 Recommended Storage Procedure for Nickel Precharged Cells/Batteries

1. Carry out a standard capacity cycle (C/10 charge and C/2 discharge) at 10°C or 20°C, with no open-circuit period between the charge and the discharge portions of the cycle. Any open-circuit stand while the cells are charged will enable charge redistribution processes in the nickel electrodes that will decrease the activity of the nickel precharge, and thus increase the probability that a hydrogen-precharged condition will be encountered.

2. Let down the cells to <5 mV with individual cell letdown resistors of at least 25/N ohm, where N is the nameplate rating of the cell in Ah.

3. Carry out a precharge evaluation using the reversal response test outlined above. Cells or batteries that have all cells with active nickel precharge may then be placed into storage in the open-circuited condition. It is recommended that this storage be at cold temperatures (5°C or less). If a battery contains even one cell that does not have active nickel precharge, the entire battery should be handled and stored as a hydrogen-precharged battery, as described in the next section.

4. If any stored cells or cells within batteries are recharged to voltage levels above 0.7 V, it should be assumed that hydrogen has been generated in the cells, and the storage preparation procedure outlined in steps 1–3 should be repeated before resuming the storage. If it can be verified that active nickel precharge remains within all cells, the storage preparation cycle in steps 1–2 is not required, and the cells may be placed directly in storage.

5. Cells that are placed in storage with verified active nickel precharge should remain in storage until they are needed. Each removal from storage to check capacity or otherwise exercise the cells increases the risk that no active nickel precharge will remain. There is no evidence suggesting that cells having active nickel precharge can spontaneously transition to hydrogen precharge during discharged storage as described above. However, over long storage periods, they can lose most active precharge such that the hydrogen generated during recharge following storage can never again be fully eliminated. This could be detected using precharge verification tests, and should result in subsequent storage being as a hydrogen-precharged cell or battery.

4.2 Recommended Storage Procedure for Cells/Batteries that have Become Hydrogen Precharged

1. Maintain all cells in a charged state. This is referred to as an active storage mode. This may be done using either a trickle charge or using a periodic top charge. Trickle charge rates should be C/100 or lower. The temperature should be kept at 62°F or less during active storage.

2. If top charging is used it should be done frequently enough to assure that all cells stay above 1.2 V. Usually a weekly top-charge is adequate. The recommended procedure for top charging is to charge at a C/10 rate until a temperature rise is seen, thus indicating some cells are in overcharge. If cells are oriented on their sides in the gravitational field, the temperature rise should be limited to no more than 1–2°C to prevent popping damage. If all cells are vertically oriented, the recharge may be continued until the temperature reaches 72°F. After allowing the cells to cool down, it is acceptable to repeat the recharge to the temperature rise limit.

3. After long periods of active storage (typically >6 months), it is possible that imbalance between the cells in a battery can develop. This may be recognized by noting whether any individual cell voltage falls below 1.27 V just prior to their scheduled top charge. This protects against the situation where cells may rapidly drop in voltage to below 1.2 V between battery monitoring intervals. The situation of significant cell imbalance can be alleviated by rebalancing the cells.

4. Batteries containing imbalanced cells can be rebalanced by several methods. If individual cell letdown is possible, rebalancing is achieved by letting all cells down to a low voltage. If individual cell letdown is not possible, rebalancing can best be achieved by battery letdown, followed by repeated recharges to the temperature rise limit, then a second cycle of battery letdown and recharge. This rebalancing process is most effective if the batteries are kept as cold as possible.

5. If individual cell voltage monitoring is not available, batteries should be rebalanced if the average cell voltage falls below 1.27 V at any time during the open-circuit stand time between top charges. This protects against the situation where cells may rapidly drop in voltage to below 1.2 V between battery monitoring intervals.

Battery storage modes other than those described above are often used because of limitations in the accessibility of the batteries, no cell monitoring, poor thermal control, or other less than optimal situations. Some of the storage alternatives that are less than optimum are described here, along with the reasons why they are not preferred,

1. *Discharged storage without a conditioning cycle in preparation for storage.* This has been used effectively, but eventually has led to problems in situations where batteries have been left partly charged and open circuited, then discharged and placed in storage. This procedure can leave significant hydrogen in cells that, if properly handled, would still have plenty of active nickel precharge.

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2. *Discharged storage without verifying precharge.* This storage method has frequently been used, either because of concerns with a brief reversal current to detect precharge, inability to monitor individual cell voltages, or because the need for verifying precharge was not fully appreciated. When cells are new and there is plenty of active nickel precharge, there is no problem. However, after 1–3 years it is not uncommon for nickel precharge to lose much of its activity, depending on handling and environment. This transition is not detected unless the precharge verification is always done prior to storage. The secondary residual capacity and voltage recovery precharge verification responses described earlier can be useful indicators in some situations to help indicate precharge.

3. *Charged open-circuit storage.* Nickel-precharged cells can be left in the partially charged state during storage without significant known adverse effects, assuming that active precharge remains after the cell completely self-discharges. This precharge will pull the platinum electrode up to the nickel electrode potential at some point during storage. While adequate active nickel precharge may be reasonably assured when a cell is new, it can become highly uncertain after a period of battery use or test. Thus, this storage mode, while simple, does entail risk of chemical damage to the nickel electrodes that increases with age and usage.

5. Summary and Conclusions

The chemical processes that are known to occur in nickel-hydrogen cells during cell or battery storage have been described for situations where the cells have either nickel or hydrogen precharge. The storage procedure that is preferred to best prevent processes that can degrade cell performance is recommended for each precharge situation. In addition, procedures are provided for verifying the precharge of cells in batteries to allow the correct storage procedure to be identified. The following general battery storage guidelines are recommended.

1. *If all cells have nickel precharge*: Discharged and open-circuited cold storage.

2. *If some cells have hydrogen precharge*: Active storage with all cells held at operating voltages.

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